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FEEDING EXPERIMENTS WITH INDIAN PEAS CONTAINING *LATHYRUS SATIVUS.*

By JOHN HUGHES.

(*Read at the Meeting, June* 5, 1895.)

WITH the assistance of Mr. H. Coleman, of Horton, near Epsom, I have recently been able to carry out, at the farm where I reside, some feeding experiments with Indian peas, as imported, containing 20 per cent. of *Lathyrus sativus* (bitter vetch).

They were purchased of a firm in Mark Lane at 18s. 6d. per quarter, and a mechanical analysis gave the following results:

Indian peas	65·41
Lathyrus sativus	20·63
Foreign seeds	10·81
Mechanical impurities (dirt)	3·15
	100·00

Separate analyses were afterwards made with the following results:

	Indian peas.	*Lathyrus sativus.*
Water (lost at 100°C.)	11·66	11·20
Oil	1·87	1·70
Albuminous compounds*	25·06	28·29
Starch, digestible fibre, mucilage, etc.	51·51	49·32
Indigestible fibre	6·97	6·73
Mineral matters†	2·93	2·76
	100·00	100·00
* Containing nitrogen	3·96	4·47
† Containing silica	0·10	0·10

The peas as delivered, containing the Lathyrus and other foreign seeds, were then ground into meal, and the following analysis represents the composition of the mixture employed for the feeding experiments:

Water (lost at 100°C.)	12·60
Oil	1·60
Albuminous compounds*	24·11
Starch, digestible fibre and mucilage	48·25
Indigestible fibre	7·97
Mineral matters†	5·47
	100·00
* Containing nitrogen	3·81
† Containing silica	2·10

It will be noticed that the mechanical impurities in the form of dirt have raised the mineral matters to 5·47, while the albuminous compounds only amount to 24·11.

The animals selected consisted of a horse between twelve and fourteen years old, and a three-year-old in-calf heifer.

Commencing on February 27, 1 lb. of the pea-meal mixed with chaffed straw, mangolds and grains, was given every morning to the cow, and 1 lb. mixed with oats and chaff to the horse, for the first fortnight.

No alteration in the general health, or in the character of the dung, was noticeable in either case.

On March 13 the daily allowance of mixed pea-meal was increased to 1½ lb. per day, both for the cow and the horse, and continued till the 27th.

On that date the horse still appeared in his usual health, but during the latter part of the second fortnight there was a slight looseness in the character of the dung, and it was decided to discontinue the pea-meal at the end of the month, and substitute 1 lb. of barley meal, which was done, and by April 3 the dung was again quite normal.

As regards the cow, there was no alteration either in her general appearance or in the dung, and on March 27 the allowance of pea-meal was increased to 2 lb. per day, and maintained regularly till April 10, thus completing a trial of six weeks.

At this date, there being no abnormal appearance in any respect, nor sign of any paralysis, the cow having improved in general health and condition, and as the object of the experiment (namely, to ascertain whether small quantities of Lathyrus *were* or *were not* poisonous) had been attained, it was decided to terminate the experiment.

There was no reason whatever why the daily allowance of the meal should not have been indefinitely continued, as far as the cow was concerned, and it should be remembered that the 2 lb. of Indian pea-meal contained 6½ oz. of Lathyrus.

All the recognised authorities state that Lathyrus only produces illness after continued or excessive use.

As regards the horse, the 1½ lb. of Indian pea-meal containing 5 oz. of Lathyrus given daily for fourteen days, appeared to have caused slight derangement of the digestive organs, the dung being loose and the animal's thirst increased, but there was neither illness nor sign of paralysis; its daily work was performed as usual, and the horse is still in excellent health.

In an alleged case of horse-poisoning at Bristol, it was stated that the horses received 2 lb. per day of Indian peas (roughly crushed) consisting chiefly of Lathyrus, and that no signs of illness appeared until after a month had elapsed.

In reporting, therefore, upon any feeding material, we should consider the quantity of the Lathyrus present, for it is the quantity that determines the quality. A cake containing 1 or 2 per cent. of sand is not likely to be injurious, but if the sand amounts to 10 or 20 per cent. the cake would very properly be pronounced unfit for feeding purposes.

It is very unfair to condemn a feeding-meal on account of the presence of a few seeds (except in the case of castor).

We should endeavour to make an approximate determination of the quantity of such seed before presuming to condemn the meal as unfit for use.

According to Dr. G. Watts, *Lathyrus sativus*, which originally was indigenous to the North-West Provinces, has spread as a weed seed all over India, being found growing not only with pulse of various kinds, but also with cereals.

Dr. J. Voelcker, in his report on "The Improvement of Indian Agriculture," at page 281, mentions that "*Lathyrus sativus* is one of the impurities found in *clean* wheat as it leaves the threshing-floors of cultivators in the Cawnpore district."

In 1888 I examined a sample of Indian horse gram and detected a foreign seed which I now recognise as Lathyrus.

Professor Leather, in his paper, in the *Veterinary Journal* for April, 1885, writes: "So far as we can learn, Lathyrus may be used with perfect safety in feeding oxen and sheep, as it does not appear to produce any symptoms in these animals."

Proportions of Husk and Kernel.

In order to determine approximately the relation between the husk, the kernel, and the whole seed, ten large and ten small seeds were respectively weighed, coarsely pounded in a mortar, and the husk carefully picked out and weighed.

	Weight of seed.	Weight of husk.	Percentage.
Ten large peas	0·895 grammes.	0·131 grammes.	14·63
Ten large peas	0·866 ,,	0·127 ,,	14·66
Ten small peas	0·254 ,,	0·048 ,,	18·89
Ten very small peas ...	0·188 ,,	0·033 ,,	17·55
Ten large Lathyrus ...	0·838 ,,	0·122 ,,	14·55
Ten large Lathyrus ...	0·812 ,,	0·119 ,,	14·65
Ten small Lathyrus ...	0·273 ,,	0·058 ,,	21·24
Ten very small Lathyrus ...	0·234 ,,	0·058 ,,	24·78

Average Composition.

	Indian pea.		*Lathyrus sativus.*	
	Large.	Small.	Large.	Small.
Kernel	85·35	81·78	85·40	76·99
Husk...	14·65	18·22	14·60	23·01

The relation of large to small being taken as follows:

	Indian pea.	*Lathyrus sativus.*
Large	48·89	64·89
Small	51·11	35·11

If, therefore, we assume that 15 parts of husk represent 100 parts of the whole seed, we shall obtain a full approximate estimate of the quantity of Lathyrus present in cake or meal.

Character and Microscopical Appearance of the Husk and Starch.

These are shown in the accompanying sheet of photo-micrographs. The testa of both seeds consists of three layers, the two inner ones being so delicate and fragile that they are usually destroyed by the chemicals employed to render the husk transparent.

The external layer is the most important for the purposes of distinction. The most convenient way to prepare it for examination is to boil alternately with acid and soda until cleared, then to thoroughly disintegrate it by grinding between a slide

and cover glass, whereby the fibres which compose it become separated and in a condition to be viewed.

With a one-quarter or one-sixth objective they are seen to be hollow throughout their whole length, and in section hexagonal at one end and stellate at the other; which explains the different appearance presented by the two sides of the husk.

So far the appearance of this outer layer on both the seeds is not very dissimilar. In the middle of the fibres of the Lathyrus, however, there is a secondary deposit of spiral tissue like a minute corkscrew, whereas in the fibre of the outer layer of the Indian pea there is no spiral tissue (see Figs. 1 and 2). Consequently a careful examination of this layer is sufficient to distinguish any isolated fragments of the husk that may be found in a feeding-cake or meal.

Of the two inner layers, the middle one is very characteristic in both seeds. In the Lathyrus it consists of a thin membrane with a strong resemblance to some patterns of muslin, being composed of numerous circular cells, which average 0·0023 inch in diameter, and which, when once observed, can easily be recognised again (see Figs. 3 and 4).

A surface view of these cells gives a very erroneous impression of their true form, for if viewed edgeways they will be seen to bear a striking resemblance to collar-studs, perforated through their length.

In the Indian pea these cells, from greater compression, lose their circular form and become hexagonal, their size being less—namely, 0·0012 inch diameter (see Figs. 3 and 4). This, together with an absence of the radial dots present in the Lathyrus, suffices to distinguish the middle layer in the respective seeds.

The third and innermost layer is exceedingly soft, is destroyed by most clearing agents, and presents no special interest for identification.

Starch.

It is not easy to differentiate single granules, but when viewed together the points of difference become apparent.

In the Lathyrus the granules vary more in size amongst themselves, and are generally rounder than those of the pea, which are more elongated, and frequently show a longitudinal depression well seen with high powers. The relative size of mature grains in the Lathyrus is 0·0018 inch diameter, and in the pea 0·0013.

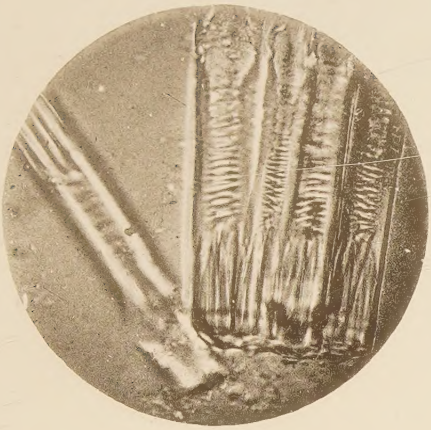
Further, the Lathyrus gives a greater play of colour with polarized light than does the Indian pea. When the Lathyrus granules are treated with a very weak solution of iodine, the blue coloration is feeble, and produced in a very irregular manner; while in the pea the coloration is much stronger and more uniform, very few granules being left unstained (see Figs. 5 and 6). The iodine solution must, however, be very weak in order to effect this distinction.

In conclusion, I have to acknowledge the valuable assistance of Mr. Albert Ashe in the microscopical work of this paper.

Discussion.

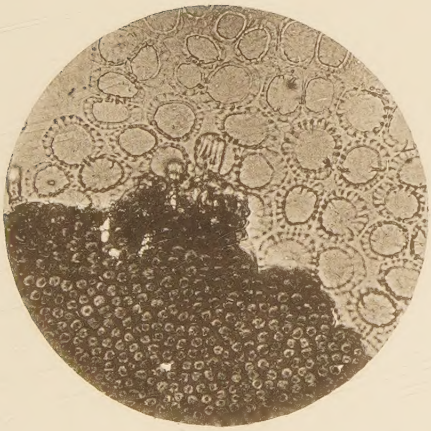
Dr. J. Augustus Voelcker said that he had never had any difficulty in distinguishing between a rounded pea like *Pisum arvense* and *Lathyrus sativus*,* which

* See Analyst, xix., 102.



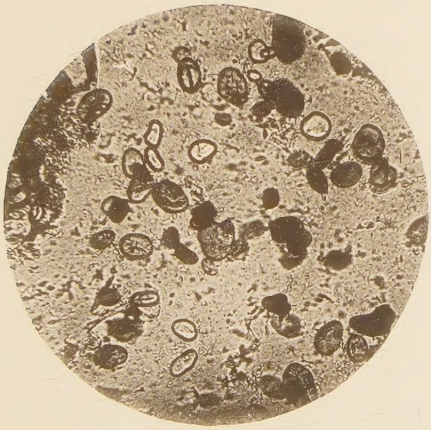
1. X 750.

2. X 600.



3. X 200.

4. X 200.



5. X 117.

6. X 117.

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was totally different in shape and appearance. There was but little difficulty in recognising *Lathyrus sativus*, even when ground up in a cake, as the husk was very characteristic. There was the ovate shape to go by, and also the peculiarly mottled or marbled appearance of the seed, which was completely absent in the common pea.

On the general question, he maintained that the most important point was not the deciding as to whether certain small quantities of *Lathyrus sativus* were in any particular case injurious. In a case where a report had to be made upon a certain small sample, it was quite impossible to give any opinion as to the nature of the whole bulk. It was known to happen frequently that, while one portion of a delivery might contain enough injurious seed to poison a whole herd, another bit from the very same delivery would be quite free from it, and the fact of only a small proportion of injurious matter being found in any particular sample was not evidence that that proportion only was maintained throughout the whole bulk.

He did not contend, and never had contended, that *Lathyrus sativus* was poisonous under *all* circumstances, but that its poisonous properties did actually exist was now recognised. The uncertainty of its action had led him to make inquiries, which had resulted in his being informed by the Director of Botany of Northern India, with whom he had corresponded, of a belief current among the natives that there were *two* kinds of *Lathyrus sativus*, one with and one without poisonous properties, though his informant was unable to say whether this was actually the case or not. The seeds of the two kinds were indistinguishable botanically, though the so-called non-poisonous kind had the seeds somewhat smaller than the other.

Mr. Hughes, in reply, said he thought it would be very difficult, if not impossible, to recognise *Lathyrus sativus* without the aid of the microscope when incorporated in a feeding meal or cake, as it would in all probability be ground very finely.

He was able to confirm what Dr. Voelcker had said as to the existence of apparently two kinds of *Lathyrus sativus*, from information which he had received from Dr. King, of the Botanical Gardens, Calcutta. The fact of the seeds being indistinguishable botanically was not surprising, as a botanist really required the whole plant for purposes of identification, the seeds alone being of but little use. It was well known that in India the crop was grown by the natives as food for themselves and their animals, in spite of the fact that they were aware of its occasionally injurious properties, which, however, they endeavoured to counteract by cooking the peas, and by using them in moderation. It is only when compelled by famine to use the seeds of *Lathyrus sativus* as the exclusive daily food that the natives experience disastrous results. Mr. Arthur Church, of the Botanical Gardens, Oxford, believes that much confusion at present exists in regard to the different species of Lathyrus, and that a more perfect identification would determine which species were dangerous and which were harmless.

PSEUDO-WOOLLEN FABRICS.

By E. G. Clayton.

(*Read at the Meeting, June* 5, 1895.)

A considerable amount of attention was directed a few months since to some published statements referring to the material known as "flannelette." In common, no doubt, with many other persons, the writer was previously under the impression that flannelette consisted of wool and cotton in about equal proportions. To a casual observer, much of the flannelette sold would seem to be softer and warmer than many flannels.

It is likely, therefore, that for some years past flannelette has been purchased frequently for the clothes of delicate persons and children, under a mistaken notion that it was the most suitable material for that purpose; instances of this are known to the writer.

The published statements alluded to above pointed to the probability that an investigation of samples of flannelette of different values, and procured at various times and places, would be of some interest.

The results of a microscopical examination of ten samples of flannelette, obtained in various neighbourhoods, and supplied at different prices, showed that six of them were all cotton, and that four of them did not contain more than 5 per cent. of wool.

Of nineteen samples of material sold as "sanitary flannel" similarly examined, sixteen were found to be all wool, one nearly all wool, one chiefly cotton, and one all cotton.

Eighteen samples of "flannel" were found to consist entirely of wool.

NOTE ON ADULTERATED SANDAL-WOOD-OIL.

By T. H. Pearmain and C. G. Moor, M.A.

(*Read at the Meeting, June 5th*, 1895.)

We have recently met with some gelatine capsules labelled and sold as "pure sandal-wood-oil capsules," the contents of which, on examination, proved to be castor-oil mixed with a small quantity of an unsaponifiable oil which we have not been able to identify. The oil had the characteristic taste and smell of castor-oil. It gave the following figures on analysis:

Specific gravity at 15·5° = ·9633.
Iodine absorption = 88·0 per cent.
Unsaponifiable matter = 5·0 per cent.
Valenta test (using 84 per cent. acid) turbid at 100° C.
Miscible with glacial acetic acid.
Miscible with 3 volumes of rectified spirit.
Saponification value = 17·0 per cent. KHO
(Corrected for unsaponifiable matter = 17·9 per cent. KHO).

Rotation for 100 mm. at 15·5° = +8·0°.
Bromide of tin test = bright green.
Fatty acids :—
Solidifying-point, 0° C.
Melting-point, 13° C.
Iodine absorption, 90·1 per cent.

The somewhat high figures obtained in the case of the iodine absorption and rotation we attribute to the presence of the unsaponifiable oil. The highest dextro-rotatory castor-oil we have examined had an activity of 4·7°.

The unsaponifiable matter gave the green colour with the stannic bromide test to a much more marked extent than the original oil, thus proving this body to be the cause of the green coloration.

The stannic bromide test is performed as follows: To three drops of the oil under examination are added three or four drops of freshly-made stannic bromide containing a little free bromine,* the mixture well stirred, and the colour produced after two or three minutes noted. Genuine sandal-wood-oil gives a blood-red colour, the mixture becoming almost dry and solid after one hour. Cedar-oil, the most common adulterant of sandal-wood-oil, gives a purplish colour. A mixture of the two does not solidify on standing.

On account of the difficulty of obtaining a sufficient quantity of material, we have been obliged to omit many estimations, the results of which would have been of interest.

We found it necessary to dilute the acetic acid before applying the Valenta test, sandal-wood-oil being miscible with glacial acetic acid. 84 per cent. acid was found to be the most convenient strength.

The following are some results obtained on some genuine sandal-wood and cedar oils:

Sample.	Oil.	Source.	Sp. Gr. at 15·5° C.	Iodine Absorption.	Rotation 100 mm. at 15·5°.	Valenta Test (84% Acid).	Bromide of Tin Test.
A	Sandal-wood	Indian	0·9815	226·0	L 13·84°	60°	Blood-red
B	,,	,,	0·9792	225·0	L 15·21°	50°	,,
C	,,	,,	0·9798	246·5	L 15·55°	52°	,,
D	,,	,,	0·9792	244·1	L 15·70°	47°	,,
E	,,	,,	0·9840	215·3	L 19·53°	—	,,
F	,,	Australian	0·9496	239·5	R 6·19°	50°	,,
G	,,	—	0·9495	228·6	R 4·82°	52°	,,
H	Cedar	—	0·9819	200·4	L 39·0°	above 100°	Purple
I	,,	—	0·9840	241·5	L 26·0°	,,	,,
J	,,	—	—	—	L 30·0°	,,	,,

* Made by allowing dry bromine to fall drop by drop on granulated tin contained in a flask kept cool by immersion in cold water, until the product has a red colour indicating excess of bromine.

NOTE ON A SAMPLE OF ADULTERATED COFFEE.

By T. H. Pearmain and C. G. Moor, M.A.

(*Read at the Meeting, June* 5, 1895.)

We have recently obtained a small sample of adulterated coffee, which we are informed is about to be placed on sale in this country. As the results obtained on examining the sample are somewhat interesting, we thought this note would be worth communicating to the society. The sample gave on analysis the following figures:

	Per cent.
Moisture	3·2
Fat	12·3
Total ash	3·4
Insoluble ash	0·7
Soluble ash	2·7
Nitrogen	2·7

A filtered 10 per cent. decoction had the same specific gravity as genuine coffee, namely, 1009·5. After some little trouble we found the sample to consist of exhausted coffee, together with ground acorns.

We were unable to isolate any appreciable quantity of caffeine from the sample. The microscopical appearance of the sample is remarkable, and would render its detection easy even if mixed in small proportion with genuine coffee. In addition to the well-known coffee structures are a number of starch-containing cells, arranged not unlike the heads of Indian corn, together with cellular membrane-containing spiral vessels, which we found to be characteristic of ground acorns.

On the Hübl Method of Iodine Absorption. Julius Ephraim. (*Zeit. für angewand. Chemie*, Heft ix., 1895, pp. 254-259.)—Since the Hübl solution, when titrated with thiosulphate, requires a much larger quantity of the latter after the addition of KI, the conclusion is arrived at that a substance capable of liberating I from KI must be present. This substance is probably a compound of iodine and chlorine formed by the action of the iodine on the $HgCl_2$. On mixing the two portions of the Hübl solution it appears to be formed at once. Thus the author obtained the following results:

25 c.c. Hübl Solution required.	$Na_2S_2O_3$. c.c.
I. Immediately after mixing:	
(*a*) Without KI	14·30
(*b*) With KI	51·10
II. After twenty minutes:	
(*a*) Without KI	14·45
(*b*) With KI	50·25
III. After four hours fifty minutes:	
(*a*) Without KI	13·25
(*b*) With KI	48·95

(1 c.c. $Na_2S_2O_3 = 0·01223$ gramme I.)

He next shows that both iodine monochloride and iodine trichloride can be titrated directly with $Na_2S_2O_3$, and proves that the latter cannot be the compound present in the Hübl solution, since results altogether discordant are obtained when it is tried with commercial oleic acid.

Hübl's requirement, that for two atoms of iodine there must be at least one molecule of $HgCl_2$, may be explained by the following equation, on the assumption that iodine monochloride is the compound formed :

$$HgCl_2 + I_2 = HgClI + ICl.$$

To make a solution corresponding to the strength of the Hübl solution this would give 16·25 grammes of ICl per litre, and this proportion is used by the author for his solution.

Since iodine monochloride as met with in commerce is seldom quite pure, it is not sufficient merely to ascertain the amount of real ICl which it contains by titration with the addition of KI, but also without, so that the amount of I which the impurities in the ICl are capable of liberating in the substance itself may be ascertained.

The method, which is carried out in the same way as the Hübl process, gave the following results :

Oil.	Iodine Number with Iodine Monochloride.	Hübl Iodine Number.
Oleic acid, I.	82·7 ...	82·84
,, II.	82·77	
,, III.	82·5	
,, IV.	82·74	
Linseed oil, I.	179·7 ...	179·9
,, II.	177·7	
Olive oil, I.	83·9 ...	84·3
,, II.	84·00	
Poppy oil	81·13 ...	81·9
Sesame oil, I.	137·1 ...	137·3
,, II.	137·1	
Earthnut oil, I.	110·6 ...	111·1
,, II.	109·7	
Castor oil, I.	83·33	
,, II.	83·09	
Almond oil, I.	96·00	
,, II.	96·29	

From these figures the author concludes that a solution of iodine monochloride in alcohol may be substituted for the Hübl solution. The advantages are that it is more readily prepared, being easily soluble in alcohol, and when made is ready for immediate use. Moreover, it can be made in much more concentrated solution, and, lastly, is cheaper. Its chief disadvantage is that it is not yet made in a state of sufficient purity, and, therefore, each fresh quantity obtained must be titrated, as mentioned above.

If the ICl solution, after acting on the oil, be titrated without previous addition of KI, a new value is obtained, which the author calls the "chlor-iodine number." In titrating, the thiosulphate is added until the liquid, which had become brown with separated iodine, again becomes yellow. The solution is then diluted, starch added, and the titration completed.

The reaction taking place is :

$$ICl + 2Na_2S_2O_3 = NaI + NaCl + Na_2S_4O_6,$$

in which 161·5 parts of ICl = 316 of $Na_2S_2O_3$. The following chlor-iodine numbers were obtained :

	I.	II.
Earthnut oil	56·6 ...	55·91
Linseed oil	77·03 ...	76·7
Sesame oil	65·3 ...	65·0
Rape-seed oil	50·4 ...	50·06
Poppy oil...	43·4 ...	43·4
Castor oil...	44·24 ...	43·88

A few experiments tried with iodine bromide also gave concordant results :

	I.	II.	III.
Almond oil	58·5 ...	58·6	
Olive oil	52·6 ...	52·81 ...	52·24
Earthnut oil	62·9 ...	63·29	

The solution of IBr contained 20·7 grammes per litre. C. A. M.

The Influence of Temperature on the Elaidin Reaction. A. P. Lidow. (*Pharm. Zeit. f. Russland*, xxxiv., 105-106, through *Chem. Centralblatt*).—By the action of nitrous oxides on oleic acid at 0° C. a substance was produced which melted at 42° C., while there was an increase in weight of 1 per cent. At 80-85° there was an increase of weight of 16·6 per cent., and the iodine number of the product was only 9 per cent., whereas that of the oleic acid used was 88·6 per cent. It thus appears that at this temperature the free affinities of the unsaturated acids are satisfied. By long-continued action of the nitrous oxides the elaidic acid formed becomes fluid. C. A. M.

Action of Alcoholic Soda Solution on Albumin and on Glue-yielding Substances. W. Fahrion. (*Chem. Zeit.*, 1895, xix., 1000-1002.)—The author has already shown (*Chem. Zeit.*, 1893, xvii., 434) that unsaturated fatty acids easily undergo polymerization, and that the polymerides are equally easily broken up by alcoholic soda solution; since albuminoids and gelatinoids have been regarded as polymerization products, it appeared to be of interest to study their behaviour with the same reagent.

Ten grammes of pure hide-powder were digested on the water-bath with 50 c.c. of alcoholic soda solution (8 per cent.), whereupon nearly all of it dissolved, a little ammonia being at the same time evolved. The alcohol was evaporated, the gelatinous residue dissolved in hot water, and the solution cooled and filtered. The filtrate was heated, acidified with hydrochloric acid—which expelled some carbonic acid and a little sulphuretted hydrogen—cooled, and filtered. The yellow filtrate was evaporated to dryness, the residue was dried at 110 to 120° until constant in weight, and extracted with hot absolute alcohol; the alcoholic solution left a sticky red-brown syrup when evaporated.

This product is nearly ash-free and quite sulphur-free; it cannot be crystallized. It has a sour odour and a disagreeable, bitter taste. It is insoluble in ether and in

light petroleum, but dissolves easily in water and in alcohol. When heated for some time it swells up, but does not apparently decompose; when cooled again it becomes brittle and easily powdered, but the powder is extremely hygroscopic. The substance is undoubtedly an acid, for it liberates carbonic acid from sodium carbonate in the cold, but its salts appear to be soluble, for its aqueous solution yields no precipitate.

An analysis of the acid, dried at 110°, indicated the formula $C_8H_{16}N_2O_6$ for the compound, but the author is disposed to regard it as still containing a molecule of water, and as being in reality identical with the acid obtained by Schutzenberger by heating albumin with baryta under pressure, and called by him proteic acid, $C_8H_{14}N_2O_5$. Towards the carbonates of the alkaline earths the acid behaves as though it were monobasic, but when it is heated with the alkaline earths themselves it behaves as a dibasic acid; this observation leads the author to conclude that proteic acid is a lactonic acid, which yields its appropriate salts when it reacts with carbonated alkalies, but salts of a dibasic acid containing one more molecule of H_2O when heated with caustic alkalies.

The same acid was obtained by the similar treatment of glue, degreased ox-flesh, casein, human hair, horn, wool, and silk, indicating that the proteids in general are not aldehydic in constitution, as supposed by Loew, but lactonic.

More important from the point of view of this journal are the possible applications of this reaction of proteids in analysis.

To distinguish wool and silk from cotton, a sample of the fabric is warmed with 8 per cent. alcoholic soda solution; wool and silk dissolve with the exception of a little dark flocculent matter, but cotton is not attacked. Starch yields a black substance when treated with the alcoholic soda solution, but this is insoluble in alcohol, so that a quantitative estimation of albuminoids in presence of starch will probably be possible by this method.

Most oils and fats contain a small percentage of albuminous substance, and when they are saponified with alcoholic soda, this albumin is converted into proteic acid, which remains dissolved. When an excess of acid is added, and the liquid is shaken with petroleum ether for the purpose of separating the hydroxy-fatty acids (*loc. cit.*), which are insoluble in this solvent, the proteic acid also remains undissolved, and can be detected in the hydroxy-acids by means of its nitrogen. It is the presence of this proteic acid which has misled Jahoda and Simand in their investigation of dégras; these chemists have maintained that a peculiar nitrogenous constituent—the "dégras-former"—is essential to genuine dégras, and can be recognised by its insolubility in petroleum ether. The author has shown, however, that by shaking the dégras with ether and water before saponification, the portion of the fatty acids which is insoluble in petroleum ether is free from nitrogen, consisting, in fact, of pure hydroxy-fatty acids. The method by which dégras is obtained necessitates the presence of a small quantity of hide-fibre, which will, of course, yield proteic acid when the dégras is saponified; thus the presence of nitrogen in that portion of the fatty acids of dégras which is insoluble in petroleum ether is not *essential* to genuine dégras, although it is indicative that the material has been obtained from chamois leather.

The author submits the following scheme for the analysis of chamois leather and of glacé kid:

Two portions of 5 grammes of the finely-divided sample are weighed; the one is dried at 110° to 120° for the estimation of water, and subsequently ashed for the determination of mineral matter; the other is saponified in a porcelain dish with 8 per cent. alcoholic soda solution; the alkaline solution is washed into a separating funnel, acidified with hydrochloric acid, and when cold shaken with ether. The solid hydroxy-acids—the higher oxidation products of the jecoric acid which is characteristic of the fish oils—are thus left undissolved; they are dissolved in warm alcohol, and, after this has been evaporated, weighed. The ethereal solution contains the unsaponifiable matter, the fatty acids, and the liquid hydroxy-fatty acids. It is evaporated, and the weighed residue is treated with petroleum ether, which leaves the hydroxy-acids undissolved. The petroleum ether solution is shaken in a separating-funnel with aqueous alcoholic soda solution; the fatty acids are thus extracted, the unsaponifiable matter (cholesterin) remaining dissolved in the petroleum ether. The alkaline solution of the fatty acids is evaporated to dryness, the residue dissolved in hot water, the solution decomposed with hydrochloric acid, and the fatty acids shaken out with petroleum ether (distilling completely at 75°).

The following table gives some analyses made according to this scheme. The small quantity of substance insoluble in the original alcoholic soda solution has been neglected, and the hide substance has been calculated by difference:

	Water, Per Cent.	Ash, Per Cent.	Unsaponifiable matter, Per Cent.	Fatty Acids, Per Cent.	Liquid Hydroxy-acids, Per Cent.	Solid Hydroxy-acids, Per Cent.	Hide Substance, Per Cent.
Chamois leather (Sheep), I. ...	18·66	8·28	0·49	4·15	0·37	0·53	67·52
,, ,, ,, II. ...	17·95	1·38	0·10	1·17	Trace	0·69	68·71
,, ,, ,, III. ...	17·60	4·38	0·30	3·10	0·28	1·00	73·34
,, ,, (Doe)	15·15	6·03	0·49	4·16	0·45	1·37	72·35
,, ,, (Goat)... ...	15·18	3·83	3·03	0·28	0·61	0·56	76·51
,, ,, (Buffalo) ...	20·54	3·21	0·10	0·46	0·11	0·56	75·02
Glacé kid...	11·69	7·12	3·66	6·58	0·66	0·75	69·54

It is particularly noticeable from these figures how very little fatty matter serves to convert skins into chamois leather. The author is disappointed to find that the proportion of fatty acids present is, as a rule, much greater than that of the hydroxy-acids, since, according to his theory, the curing of the hide-fibre depends on its combination with hydroxy-fatty acids; he has not yet, however, satisfied himself that the fatty acids are not merely derived from the natural fat of the skin.

A. G. B.

The Examination of Pepper. W. Busse. (*Mitth. Gesundheitsamt.*, 505, through *Zeit. angewand. Chem.*)—In the author's opinion most of the analytical methods for the valuation of pepper, such as the determination of the moisture, ethereal oil, mineral matter, alkalies, and phosphoric acid, are inconclusive, since these constituents are common to the seed and the husk. The value of the cellulose and dextrose estimations must also be called in question. On the other hand, the brown colouring substances are only found in the husk, and their quantitative estimation

will show the value of the pepper. To isolate them the following method is proposed. Five grammes of the sifted and dried pepper are treated with boiling absolute alcohol. The residue, after being freed from alcohol in the drying-oven, is ground up with a little water in a basin, and then washed into a flask with 50 to 60 c.c. of boiling water. Twenty-five c.c. of a 10 per cent. solution of soda are added, and the flask is warmed on the water-bath for five hours with constant shaking. Concentrated acetic acid is next added, until the liquid is only feebly alkaline, then 250 c.c. of water, and the flask is well shaken. After twelve hours the liquid is filtered with the aid of a suction-pump.

To 50 c.c. of the filtrate concentrated acetic acid is added to acid reaction, followed by 20 c.c. of a 10 per cent. solution of lead acetate in dilute acetic acid. After mixing, the liquid is diluted to a 100 c.c. with water, well shaken and filtered. Ten c.c. of the filtrate are decomposed with 5 c.c. of H_2SO_4 (1 : 3) and 30 c.c. of alcohol, the precipitate filtered after some time, washed with alcohol, and the lead sulphate ignited and weighed in the usual manner, and the amount of lead calculated.

The amount of lead (in grammes) which has been obtained by the process from 1 gramme of dried pepper, may be described as the "lead number." The figures given by the different kinds of pepper are :

	Lead Number.
White pepper	0·006 to 0·027
Black pepper	0·054 ,, 0·075
Husks	0·129 ,, 0·157
Pepper dust	0·109 ,, 0·122

The author concludes that this method, taken in conjunction with the determination of the ash and sand and the microscopical examination, should be of great assistance in estimating the value of pepper. C. A. M.

Simple Method of Detecting "Poivrette" in Ground Pepper. D. Martelli. (*Staz. Sper. Ag. Ital.*, xxviii., 53.)—Several methods other than microscopical ones have been proposed for the detection of "poivrette" (ground olive-stones). Thus, Gillet (*Bull. Soc. Chim.*, 50, 173) used tincture of iodine (6·5 gr. I in 120 c.c. 90 per cent. alcohol), which coloured pepper brown or dark chestnut, while olive-stones became bright yellow. Chevreau (*Rep. Pharm.*, 1889, p. 203) does not consider this reliable, and employs a mixture of 1 part aniline with 3 parts acetic acid, which colours "poivrette" a light yellow. Jumeau (*Journ. Pharm. Chim.*, 1889, p. 442) used a mixture of 5 grammes iodine in 100 c.c. of a mixture of alcohol and ether, the colours produced being the same as with Gillet's test. Pabst (*La Pratique des Essais Commerciaux—Matières Organique, Halphen*, p. 21) tests by adding a little of a solution of dimethyl-paraphenylene-diamine; after a short time the ligneous particles foreign to pepper become carmine-red, while pepper at most is superficially tinged pink.

The author proposes the following test: Digest for two or three days 1 gramme of phloroglucol in 50 or 60 c.c. of hydrochloric acid sp. gr. 1·1, and decant the clear solution. To about ½ gramme of the sample of pepper add enough of the reagent

to cover it, and heat cautiously till fumes of hydrochloric acid begin to come off. "Poivrette" and like substances (*e.g.*, ground-up shells of almonds, walnuts, nuts, etc.) give a very intense cherry-red colour, which is sharply distinguished by the naked eye from the yellow or faintly red-brown colour of the pepper. On adding to the mass a little water, and decanting the liquid, a violet-red powder is left, which consists almost entirely of "poivrette," etc., stained by the reagent.

H. D. R.

A Comparative Examination of the Constitution of Different Meat Extracts. **A. Stutzer** (*Zeit. angew. Chem.*, 1895, p. 157).—The meat extracts most frequently met with are those of the Liebig and Kemmerich Companies and the various Bovril preparations. According to the author's analysis, these contain :

	Liebig Extract.	Kemmerich Extract.	Bovril Fluid Beef.	Bovril Fluid Beef seasoned.	Bovril for Invalids.	Bovril Beef Jelly.	Bovril Lozenges.
	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
Water	17·72	16·54	29·14	44·42	28·13	89·15	9·47
Sodium Chloride ...	3·11	4·15	14·12	10·72	4·57	0·26	1·63
Other Salts	19·63	17·96	3·38	7·60	11·50	1·04	5·71
Organic Matter ...	59·54	61·35	53·36	37·26	55·80	9·55	83·19

The most important constituents of the organic matter are those containing nitrogen. Nitrogen was present in the form of—

(*a*) Albumose Peptone	0·56	1·24	1·23	0·34	1·26	0·16	2·06
(*b*) Pancreas Peptone...	2·72	2·38	3·36	1·39	3·36	0·48	6·06
	3·28	3·62	4·59	1·73	4·62	0·64	8·12
(*c*) Flesh bases and decomposition products soluble in alcohol ...	4·05	3·69	1·06	1·16	1·78	0·21	0·55
(*d*) Ditto, insoluble in alcohol	1·34	1·25	1·16	0·89	0·82	0·20	1·16
	5·39	4·94	2·22	2·05	2·60	0·41	1·71
(*e*) Albumin	0·12	0·09	0·31	0·08	0·24	—	0·42
(*f*) Muscular Fibre ...	—	—	0·73	0·90	0·70	—	0·57
	0·12	0·09	1·04	0·98	0·94	—	0·99
(*g*) Gelatin	0·04	0·05	0·09	0·09	0·15	0·29	0·70
(*h*) Ammonium Salts ...	0·48	0·46	0·31	0·27	0·38	0·12	0·42
	0·52	0·51	0·40	0·36	0·53	0·41	1·12
Total Nitrogen... ...	9·31	9·16	8·25	5·12	8·69	1·46	11·94

With regard to this table, the author remarks :

I. The value of a meat extract as a food material depends on the amount of peptone present; and of the two kinds referred to under (*a*) and (*b*), the albumose

peptone probably possesses greater nourishing power than the other. Calculating for 1 part of nitrogen $6\frac{1}{4}$ parts of peptone, the amount of the latter present in the various preparations is as follows:

	Per cent.
Liebig's Meat Extract	20·50
Kemmerich's Meat Extract	22·62
Bovril Fluid Beef	28·68
,, ,, (seasoned)	10·81
,, for Invalids	28·87
,, Beef Jelly	4·00
,, Lozenges	50·75

Thus the statement that 1 part of bovril fluid beef seasoned contains a greater amount of nourishing material than 50 parts of any other meat extract is quite inadmissible.

II. The value of a meat extract as a stimulant (apart from the action of the salt) depends especially on the quantity of the substances classed under (*c*) and (*d*). In this respect the Liebig and Kemmerich preparations take the first place.

III. In addition to albumin, the bovril preparations contain considerable quantities of muscular fibre (up to 6 per cent.).

IV. Gelatin should be regarded as a worthless constituent, and should be removed from meat extracts as completely as possible.

C. A. M.

Determination of Glycerol in Wines, and Indirect Estimation of Mannitol. G. Mancuso-Lima and G. Sgarlata. (*Staz. Sper. Ag. Ital.*, xxviii., 236.)—The authors find that the process of Oliveri and Spica (*Gazzetta*, xx., 734), who distil the glycerol under reduced pressure at 200°, and titrate with $\frac{N}{10}$ permanganate, too long and tedious when many samples are to be examined.

They base their process on the fact that ammoniacal lead hydroxide, recently prepared, completely precipitates both glucose and mannitol, and that the glycerol can be titrated by permanganate in the filtrate after removing the lead and adding sulphuric acid.

1 c.c. of normal permanganate is equal to ·01 gramme glycerol, ·0081 gramme glucose, and ·0089 gramme mannitol.

The direct estimation of glycerol is performed by concentrating 25 c.c. of the wine to a syrup on the water-bath, and precipitating in the cold by ammoniacal hydroxide of lead (obtained by adding a slight excess of basic acetate of lead, and then an excess of ammonia); the liquid is filtered rapidly through a filter, contained in a funnel, over the top of which is placed another funnel connected with a tube containing caustic potash, and the filtrate and washings collected in a beaker, acidified with concentrated sulphuric acid, and the sulphate of lead separated by filtration; the solution is brought to the boil, and titrated hot with normal permanganate.

By precipitating with basic acetate of lead, and proceeding as before, the glucose and mannitol (if present) are left in solution, and are titrated with the glycerol with permanganate; by estimating the glucose by Fehling's solution, and subtracting the amount of permanganate equal to this and to the glycerol, from the total used, the remainder is equivalent to the mannitol. H. D. R.

The Detection of Salicylic Acid in Wine. M. Spica. (*Staz. Sper. Ag. Ital.*, xxviii., 246.)—The reaction between ferric chloride and salicylic acid, after shaking out with ether, will not detect less than ·003 gramme per litre; ether, chloroform, and carbon bisulphide all extract tartaric acid, tannin, and acetic acid from the wine, which interfere with the salicylic-reaction, and render the test less delicate. By evaporation of the ether-extract, and extraction of the residue with petroleum ether, the test is more delicate, ·002 gramme being detected.

The author heats the ether extract with nitric acid, by which means the salicylic acid is transformed into trinitrophenol (picric acid). He proves that genuine wines give no extract to ether from which picric acid is formed, and that saccharine (Fahlberg) does not yield picric acid. The delicacy of the reaction is such that ·0002 gramme salicylic acid per litre can be detected.

The test is performed thus: 10 c.c. of wine are acidified with a few drops of hydrochloric acid, and shaken with an equal volume of ether; half the ether is removed and filtered (this is not absolutely necessary); the ether is evaporated, and one drop of nitric acid is added, and the mixture cautiously heated over a flame; a few drops of ammonia are added, and then 1 c.c. of water and a piece of wool, previously treated with potash solution, and then with ether to remove fat; the solution is warmed, and the wool, after washing and drying between blotting-paper, is examined; it is dyed yellow, if salicylic acid is present. By comparison with a set of standard wools, prepared from wines to which known quantities of salicylic acid have been added, an estimation may be made. H. D. R.

Estimation of Potassium Sulphate in Wine. L. Hugouneng. (*Journ. Pharm. Chim.*, 1895 [6], i., 349; through *Chem. Zeit.*)—The author notes that the percentage of sulphuric acid (weighed as barium sulphate) found in a wine depends on whether the estimation has been made in the wine itself or in the aqueous extract of the ignited total solids. The ash yields a higher percentage than does the wine itself. This discrepancy is probably due to the existence of sulphur compounds other than potassium sulphate in the wine, which become potassium sulphate when the residue is ignited. It will be found that this difference between the sulphuric acid in the wine and in the ash of the wine is characteristically high (0·2 gramme per litre) in wine from dried fruits, a fact which proves useful in the diagnosis of a particular sample. A. G. B.

The Determination of Tannin in Wine. A. Vigna. (*Staz. Sper. Ag. Ital.*, xxviii., 19.)—Two modifications of the Carpené-Lowenthal method (precipitation with ammoniacal zinc acetate, heating to drive off excess of ammonia, and titration of the precipitate after dissolving in sulphuric acid by permanganate) are proposed. In the first, 25 c.c. of an 8 per cent. solution of zinc acetate are added to 50 c.c. of wine, and neutralized exactly with dilute ammonia, and the precipitate collected on a plain filter and washed four times with cold water. The filter is then placed on a glass plate, and the precipitate washed off by a stream of water from a wash-bottle; 20 c.c. dilute sulphuric acid (1 part acid to 4 parts water) and 20 c.c. of indigo

solution are added, and the titration is performed in the usual manner with permanganate. In the second modification, 40 c.c. of an 8 per cent. solution of potash alum are used in place of the zinc acetate, the remainder of the process being the same as before.

The author shows that both modifications give the same results as the Carpené-Lowenthal method with wines, and theoretical results with pure tannins. He prefers the alum method, as being the more expeditious. H. D. R.

Detection of Fluorine in Wine. G. Nivière and A. Hubert. (*Monit. Scient.*, 1895 [4], ix., 324; through *Chem. Zeit.*)—100 c.c. of the wine are made feebly alkaline with ammonium carbonate, the solution is boiled, and 2 to 3 c.c. of a 10 per cent. solution of calcium chloride are added. After boiling for a few minutes, the liquid is filtered, the filter ashed, the ashes mixed with a third of their weight of *precipitated* silica, and the mixture transferred to a test-tube, together with a mixture of equal parts of fuming sulphuric acid and concentrated sulphuric acid (66° Bè). The test-tube is fitted with a cork, which carries a U-tube 4 millimetres in diameter, and having three bulbs (7 to 10 millimetres diameter) blown in it. In the centre bulb one drop of water is placed. The test-tube is now heated, whereupon any fluorine will be evolved as silicon tetrafluoride, which will be decomposed by the drop of water, yielding the usual gelatinous silica and solution of hydrofluosilicic acid. In the event of mere traces of fluorine being present, no visible separation of silica in the water-drop will be noticed; in this case the U-tube must be washed out with alcohol and the washings evaporated to dryness, when the silica will be detected. A blank test must be made with the filter-paper, and great stress must be laid upon the fine state of division of the silica and upon the prescribed strength of the sulphuric acid.

For quantitative estimations the U-tube is washed with water, the washings are filtered, and to the filtrate is added a solution of potassium acetate in dilute alcohol; the precipitated potassium silicofluoride is filtered, washed with dilute alcohol (1 : 1), dried, and weighed. The factor 0·5177 will convert the weight into the corresponding weight of fluorine. One litre of wine will be necessary for a quantitative determination. The author has detected the fluorine corresponding with 1 gramme of ammonium fluoride in one hectolitre of wine with rapidity and certainty by this method. A. G. B.

Detection of Fluorine in Beer. R. Hefelmann and P. Mann. (*Pharm. Centr.-H.*, 1895, xvi., 249; through *Chem. Zeit.*)—A fluoride which has been added to finished beer as a preservative may be detected as follows: To 500 c.c. of the beer, freed from carbonic acid—either by exposure in a thin layer, or by heating at 40°—1 c.c. of a mixture of equal volumes of calcium chloride solution (10 per cent.) and barium chloride solution (10 per cent.) is added; this is followed by 0·5 c.c. of acetic acid (20 per cent.) and 50 c.c. of alcohol (90 per cent.). The liquid is allowed to remain for twenty-four hours in the cold, in order that the precipitated calcium fluoride and barium silico-fluoride may settle; it is then filtered through a small filter, the last traces of the precipitate being either washed out of the beaker by

means of the filtrate, or wiped out by means of filter-paper. The precipitate and filter are dried without being washed, and transferred to a platinum crucible (20 c.c.); 1 c.c. of strong sulphuric acid is added, the crucible is covered with a clock-glass (which has been waxed and then marked with a style), this is filled with water, and the crucible is heated at 100° for two hours. By this method so little as 0·7 milligrammes of fluorine in 100 c.c. of beer can be detected with certainty.

A. G. B.

The Detection of Bloodstains in Forensic Cases. F. Gantter. (*Zeit. für anal. Chem.*, 1895, *Zweites Heft*, pp. 159-160.) — The detection of bloodstains on rusty iron is a matter of extreme difficulty, owing to the fact that in most cases it is impossible to obtain hæmine crystals from the blood which has been made insoluble by the iron oxide. Very frequently, too, it is of the greatest importance to prove the complete absence of blood.

The behaviour of the blood towards hydrogen peroxide is a valuable means for the latter purpose. The slightest trace of blood-substance brought into contact with a drop of this reagent causes an immediate evolution of oxygen, so that the drop in a very short time becomes changed into a white froth.

The best method of observing the reaction is as follows: A drop of the solution of the suspected blood-material, or where a solution is not possible (as in the case of spots on rusty iron), a small piece of the rust scraped off, is placed on an object-glass with a piece of black paper underneath. After moistening with a drop of water made very feebly alkaline a drop of hydrogen peroxide is added, and when the slightest trace of blood-substance is present numerous comparatively large bubbles of gas are developed, which after a short time unite, forming a fine snow-white scum, which remains for some hours. It is characteristic that the scum forms from the outside of the drop inwards, so that it is surrounded by a ring of clear fluid. In the case of rust containing blood, the development of gas does not proceed from all the particles of rust but only from those to which blood is attached.

It is impossible to mistake this appearance for that formed by air-bubbles. On moistening the rust with the alkaline water numerous air-bubbles are frequently formed, but these are quickly dissipated by touching them with a thin glass rod before adding the hydrogen peroxide.

If the reaction does not occur, it is certain that the rust contains no blood; but, unfortunately, its occurrence cannot be regarded as proof positive of the presence of blood, since other animal fluids (*e.g.*, pus) behave in a similar way towards hydrogen peroxide.

Apart from its value as a negative proof, the reaction is also of use in testing whether crystals supposed to be hæmine crystals really are so. The age of the spots on the rust appears to make no difference. Stains six months old responded to the test as sharply as when fresh.

C. A. M.

Examination of Medicaments containing Iodine. G. Deniges. (*Journ. Pharm. Chim.*, 1895 [6], i., 354; through *Chem. Zeit.*)—Since a large number of organic iodine preparations easily yield their iodine as alkali-metal iodide, it is possible

to assay them by the usual methods for titrating iodine. Iodoform is decomposed in a test-tube by heating it for a short time with 1 c.c. of nitric acid and 2 c.c. of hydrochloric acid; 10 c.c. of water are added and 2·5 c.c. of sodium bisulphite solution (36·4° B). The iodine having been thus completely converted into hydriodic acid, 10 c.c. of ammonia are added, and the whole is made up to 100 c.c. Fifty c.c. are then mixed with 15 c.c. of ammonia and 25 c.c. of $\frac{N}{10}$ silver nitrate solution, made up to 100 c.c. and filtered. Fifty c.c. of the filtrate are mixed with 50 c.c. of water and 10 c.c. of $\frac{N}{10}$ potassium cyanide solution; a few drops of potassium iodide solution (20 per cent.) are next added, and the liquid titrated with $\frac{N}{10}$ silver nitrate solution until a permanent turbidity is produced.

Di-iodoform is decomposed by heating it in a long test-tube with 1 c.c. of pure sulphuric acid until no more iodine vapour is evolved. The iodine will condense on the sides of the tube, and, after the addition of a few drops of nitric acid, is converted into hydriodic acid by sodium bisulphite. Aristol is decomposed by heating 0·125 gramme with 2·5 c.c of alcoholic potash (10 per cent.) and 0·5 gramme of powdered potassium nitrate, until the mass is dry and ignited. The residue is dissolved in water, reduced with sodium bisulphite and titrated as described above.

A. G. B.

On the Estimation of Sulphur in Petroleum. Fr. Heusler. (*Zeit. für angewand. Chemie*, 1895. Heft 10, pp. 285-286.)—Several of the methods for estimating sulphur in mineral oils (*e.g.*, those of Kast and Lagai. *Dingl.*, **284**, 69, and Heusler, *Ber.*, **28**, 493) cannot be relied upon when the amount to be determined is very small—0·02 gramme and less.

In his handbook of "Chemical Technology," Fischer describes a process for estimating sulphur in coal-gas, which consists in conducting the products of combustion through a tube bent many times, in which the sulphur is oxidized by a solution of hydrogen peroxide.

The principle of this method has been adapted by the author to the estimation of small quantities of sulphur in mineral oils. His apparatus consists of a small petroleum lamp, to be placed under a cylinder with an opening in the side near the bottom for air to enter, and an opening at the top for the gases to pass off. By means of a special contrivance, which cannot be clearly described without the illustration, the sulphur in the products of combustion is oxidized by a solution of potassium permanganate, which drops from a spiral worm, meets the gas, and then falls into a small flask.*

In making the estimation, 20 to 30 grammes of the oil are placed in the lamp, which is then weighed, lit, and placed under the cylinder. By means of an aspirator at the top a current of air is drawn through the apparatus, and is so regulated that the combustion is complete. The lamp may then be left burning for twelve hours or

* The complete apparatus may be obtained from C. Gerhardt, Marquart's Lager Chemischer Utensilien, Bonn.

longer. At the end of the experiment it is again weighed, and the weight deducted from the previous weight gives the amount of oil consumed. The permanganate solution is boiled with HCl and filtered, and the sulphuric acid precipitated with $BaCl_2$. C. A. M.

Estimation of Thiophen in Benzene. G. Deniges. (*Compt. rend.*, 1895, cxx., 781; through *Chem. Zeit.*)—This can be effected by precipitating the thiophen in the form of the compound $2(HgO,HgSO_4),C_4H_4S$, as follows: Two c.c. of the benzene are added to 20 c.c. of a solution of basic mercuric sulphate (50 grammes HgO, 200 c.c. H_2SO_4, 1,000 c.c. water) contained in a well-stoppered flask (60 c.c.). The stopper is wired down, and the flask heated in a water-bath for about a quarter of an hour. When cold the liquid is filtered through a weighed filter, which is then washed with hot water, dried at 110-115°, and weighed. The increase in the weight of the filter is multiplied by the factor 0·0758 to obtain the weight of thiophen in the sample.

If objection be taken to the heating of the flask, this may be avoided by dissolving the benzene in some solvent miscible with water. A freshly-prepared mixture of 10 c.c. of the mercuric sulphate solution with 30 c.c. of acetone-free methyl-alcohol will dissolve 4 c.c. of pure benzene to a clear solution; but if thiophen be present a precipitate will form in a short time, and may be weighed in the manner described above. Care must be taken that the basic mercuric sulphate is in excess, otherwise the compound $2HgO,HSO_4,C_4H_4S$ is likely to be formed. A. G. B.

Luteol; A New Indicator. W. Autenrieth (*Arch. Pharm.*, 1895, ccxxxiii., 43; through *Chem. Zeit.*) — Luteol or chlorohydroxydiphenyl-quinoxaline is obtained from phenacetin. It crystallizes in slender yellowish needles, melts at 246°, is insoluble in water and sparingly soluble in alcohol, and can be sublimed It serves as an indicator in those cases where litmus or phenolphthalein is inapplicable. In acid liquids it is perfectly colourless, but intensely yellow in alkaline solutions.

A. G. B.

The Composition of some "Vegetable" Colouring Matters for use in Confectionery, etc. G. Posetto. (*Zeits. Nahrungsm. Unters. u. Hygiene*, 1895, ix., 150; through *Chem. Zeit. Rep.*, 1895, 188.)—A set of these "vegetable" colours, consisting of pastes mostly insoluble in water and alcohol, have been examined and were found to be tin lakes. When dry they contain from 72 to 80 per cent. of tin oxide, and as in their moist condition they contain about 70 per cent. of water, the amount of tin present must be at least 20 per cent. The blue paste is pure indigo-carmine, with 2·5 per cent. of ash, chiefly sulphates.

F. H. L.

A Characteristic Reaction of Citric Acid. L. Stahr. (*Nordisk pharm. Tidskrift*, 1895, ii., 141; through *Chem. Zeit. Rep.*, 1895, 187.)—If 10 mgrms. of citric acid be dissolved in 1 c.c. of water, a few drops of $\frac{N}{10}$ permanganate added and the

liquid warmed till the colour has vanished, and 3 to 5 drops of saturated bromine water dropped in, on cooling, a white crystalline precipitate appears, which, on the addition of caustic soda, yields the odour of bromoform. The reaction will detect 0·2 mg. of citric acid.

F. H. L.

The Estimation of 'Available' Phosphoric Acid in Thomas Slag. G. Sani. (*Staz. Sper. Ag. Ital.*, xxviii., 275.)—The author takes 5 grammes of the slag, adds 100 c.c. of water, and saturates at a boiling temperature with 10 per cent. citric acid solution, adds 200 c.c. of ammonium citrate solution; the mixture is then heated for an hour on the water-bath, with careful shaking, and made up to 500 c.c. The phosphoric acid dissolved is then estimated in the usual manner. H. D. R.

The Methods of Analysis of Copper Salts. L. Sostegni. (*Staz. Sper. Ag. Ital.*, xxviii., 167.)—The author shows that in the electrolytic method of determination of copper, arsenic, if present, is deposited at the same time. This method is too long, and requires too much attention.

Precipitation with zinc gave slightly high results, due to the deposition of a basic copper salt, and possibly, also, of cuprous chloride.

Titration with sodium sulphide, using as indicator paper prepared with alkaline lead acetate, was found to be inaccurate. The method of titration with ferrocyanide, using paper moistened with ferric chloride solution as indicator, was not found to be delicate enough for commercial purposes.

One gramme of the copper sulphate to be tested is dissolved in 25 c.c. of water and 15 c.c. of Soxhlet's alkaline tartrate solution added, and into this solution 20 c.c. of a 1 per cent. solution of glucose are dropped, little by little, the whole being boiled, and the boiling continued for five minutes. 25 c.c. of boiling water are now added, and after allowing the cuprous oxide to settle for a short time, the solution is decanted through a filter; boiling water is at once poured on the cuprous oxide, and the filter is then well washed before the precipitate is transferred to it. The cuprous oxide is then transferred to the filter, washed till the water which runs through is neutral, and the filter and its contents dried in a stove. The bulk of the precipitate is transferred to a platinum boat, the filter burnt and placed at one end of the boat, the whole then transferred to a tube, a piece of asbestos-paper being placed under the boat, and the Cu_2O reduced in a current of hydrogen. The results are very good, and iron does not interfere if present in traces. If notable quantities are present, the iron should be brought to the ferric state and precipitated by ammonia.

The cuprous oxide may be also converted into cuprous sulphide, and weighed as such, by placing a little sulphur in the boat (which should then be of porcelain) before placing it in the tube, and proceeding as before. H. D. R.

Estimation of Tellurium in Copper Bullion. C. Whitehead. (*Jour. Amer. Chem. Soc.*, xvii., 1895, 280-281.)—From 25 to 50 grammes of the bullion are

dissolved in HNO_3 of 32° Bé, and the excess of acid removed by boiling. To this solution ferric nitrate solution, corresponding to 0·25 gramme of iron, is added. Then an excess of ammonia, and the precipitate is washed with dilute ammonia until all the copper salts are completely removed. If necessary, the precipitate is dissolved and re-precipitated. The residue on the filter, consisting of iron tellurite and selenite, is dissolved in HCl, an excess of tartaric acid added, the solution made alkaline with KOH, and H_2S passed through, when the selenium and tellurium dissolve as sulphides. The liquid is filtered, dilute HCl added to decompose the alkaline sulphides, and after standing in a warm place to remove H_2S, again filtered. The tellurium and selenium sulphides are dissolved in aqua regia, the solution evaporated to dryness, then taken up with HCl, and the tellurium and selenium precipitated by a current of SO_2 passed in to saturation. After standing twelve hours in a warm place, the precipitate is filtered on a weighed filter, dried at 100°, and the tellurium and selenium weighed together. The selenium is separated by boiling the precipitate in concentrated KCN solution, filtering, and adding HCl, when the selenium is deposited as a brick-red precipitate, which is dried at 100° and weighed.

The tellurium is estimated by difference, or the small amount dissolved by the KCN solution may be precipitated by SO_2, after removal of the selenium, added to the original residue, and the whole dried at 100° and weighed.

C. A. M.

The Estimation of Antimony as Antimonious Antimoniate. Otto Brunck. (*Zeit. für anal. Chem.*, 1895, *Zweites Heft*, pp. 171-174.)—This method of estimating antimony has for some time past been in disrepute, since Bunsen showed that the tetroxide was volatile on ignition, and lost as much as 0·1 gramme in six hours. This observation being opposed to the general statements that Sb_2O_4 is infusible and non-volatile led the author to investigate the method.

Antimony, obtained pure by reducing potassium antimoniate with potassium cyanide, was weighed into a porcelain crucible and oxidized with HNO_3, the crucible being covered with a watch-glass. The contents were then ignited in the crucible without a lid until constant in weight, and the amount of antimony calculated from the Sb_2O_4 found. In six determinations the amount found corresponded to within a milligramme of that taken. The uncovered crucible could be kept at a red heat for several hours without losing the slightest amount in weight.

Experiments were then made to discover under what conditions a loss by volatilization could occur, and it was found that the decomposition of the Sb_2O_4 into the trioxide and oxygen admitted of a simple explanation. Where the crucible was covered with a lid the flame gases were caught by its projecting edge, conducted into the crucible, and exercised a reducing action there. When the interior of the crucible had free contact with the atmospheric air, this reducing action was altogether obviated.

With the precaution of igniting the residue in an uncovered crucible the method is not only very rapid and exact, but where small quantities of antimony are to be estimated is to be preferred to any other.

C. A. M.

The Superiority of Barium Hydroxide Solution as an Absorbent in Carbon Determinations in Steel. J. O. Handy. (*Journ. Amer. Chem. Soc.*, 1895, xvii., pp. 247-251.)—The absorption of CO_2 by means of KOH in Geissler bulbs has the disadvantage that variations of the temperature and degree of moisture in the air of the laboratory produce errors which the greatest care can only partially eliminate By using barium hydroxide instead of potash these errors are avoided, and it is possible to finish the analysis in at least three ways. 1. The barium carbonate may be filtered off, washed, ignited, and weighed. 2. The excess of barium hydroxide may be titrated (*a*) with standard oxalic acid without filtering; (*b*) with standard H_2SO_4 after filtering off the barium carbonate. Phenolphthalein is used as the indicator in each case, but the end reaction with H_2SO_4 is sharper.

In the estimation, 10 grammes or more of a steel containing 0·06 per cent. carbon, or 5 grammes of one containing 0·20 per cent., are dissolved by shaking in a stoppered bottle with a mixture of copper and potassium chloride solution (1 lb. in 1,400 c.c. of water) and HCl. 600 c.c. of the former and 40 c.c. of the acid are used for 10 grammes of steel, and proportionately more or less for other amounts. The liquid is filtered through asbestos which has been ignited in oxygen, and the carbon residue dried at 100°, and burned in the usual way in the combustion tube. The products of combustion pass through a U-tube with $CaCl_2$ in the first limb and anhydrous copper sulphate in the other, and then through two absorption tubes, each containing 50 c.c. of barium hydroxide solution. When the combustion is complete, and pure air or oxygen has been passed through to expel all carbon dioxide, the absorption tubes are disconnected and their contents filtered. During filtration air free from CO_2 is kept playing on the surface of the barium hydroxide solution, to prevent further formation of carbonate. The precipitate is washed with distilled water free from CO_2, and the excess of barium hydroxide titrated with decinormal H_2SO_4, of which 1 c.c. = 0·0006 gramme carbon. The barium hydroxide solution used contains about 20 grammes per litre.

The following results were obtained in experiments to test the method.

	KOH Method. Chemist "L," "C."	$Ba(OH)_2$ Method. Chemist "H."	Colour Test. KOH Standard.
Steel "A" Carbon	0·14	0·134	0·14
,, 11834·7 Carbon	0·109	—	0·11
,, 11857·0 ,, ...	0·178	—	0·17
,, "M" ,, ...	0·08	{0·090 and 0·093} {0·091 and 0·092}	0·08

C. A. M.

Lead Carbonate as an Agent for opening up Silicates. P. Jannasch. (*Zeits. Anorg. Chem.*, 1895, viii., 364; through *Chem. Zeit.*)—The silicate is fused with pure lead carbonate in a platinum crucible. The melt is loosened by plunging the hot crucible in cold water, and is dissolved in strong nitric acid. The silica is separated by evaporation to dryness, and the lead separated from the filtrate; on the addition of HCl, most of the lead separates as chloride, and the remainder is removed as sulphide. The lead carbonate is best prepared by precipitating lead acetate with ammonium carbonate, and washing with hot water. A. G. B.

CORRESPONDENCE.

To the Editors of THE ANALYST.

The Yews, Reigate, *July* 11, 1895.

SIRS,—Your issue for this month contains the report of a paper read by Mr. W. C. Young, F.I.C., F.C.S., at the meeting of the Society of Public Analysts, on May 1 last, on "A Comparison of the Organic Carbon and Nitrogen Results obtained by Dr. Frankland and the Companies' Analysts from the Waters supplied by the Metropolitan Water Companies," and animadverting on my method of determining organic carbon and organic nitrogen.

Mr. Young does not seem to be aware that the cause of the discrepancies which he mentions had been already explained in my annual report to the Registrar-General for 1894, published in his "Annual Summary" for that year. As this "Annual Summary" is probably not extensively read by analytical chemists, I will thank you to insert in your next number the following extract, which has reference to the matter discussed in Mr. Young's paper:

"The relation between the amounts of organic carbon and organic nitrogen recorded in these tables affords data from which an opinion may be formed as to the origin of the organic matter, whether animal or vegetable. If the relative proportion of nitrogen to carbon be high, the inference is that the organic matter is chiefly animal; on the other hand, if it be low, it is certain that the organic matter is chiefly, if not entirely, of vegetable origin. Examined from this point of view, these tables indicate that the organic matter present in the river waters as delivered in London was, to a very large extent, of vegetable origin. In reference to this subject, the attention of the Local Government Board was recently called by the London County Council to the discrepancy between my results, as given in these tables in my former reports, and those furnished by the chemists who make analyses for the Metropolitan Water Companies, the results obtained by the companies' chemists indicating that the organic matter present in the waters was chiefly of animal origin, whilst, as just mentioned, my analytical results assigned to it chiefly a vegetable origin. At a consultation with these chemists in the month of June last it was discovered that, owing to an error in the method of analysis pursued by the companies' chemists, they had obtained results which showed too large a proportion of nitrogen to carbon in the organic matters. There had also been for a long time a considerable discrepancy between our analytical results in the case of the Chelsea Company's water, the cause of which was discovered at the same time. The assistant who collected the samples for the companies' chemists had, during two years, inadvertently taken samples of the New River Company's water, believing them to be drawn from the Chelsea Company's main. Consequently, the results reported as having been obtained by them with the Chelsea Company's water had, in fact, been got in the analyses of water drawn from the New River Company's mains. These errors were at once remedied, and since that time our analytical results have been in close accordance."

I will only add here, for the special information of analytical chemists, that the chief error in the method of analysis pursued by the companies' chemists consisted in the omission of an inch of oxide of copper in front of the copper gauze in the combustion tube. This omission causes the admixture of a small quantity of carbonic oxide with the gases resulting from the combustion; and, unless a special determination of carbonic oxide is made, this gas would of course be measured as nitrogen; thus increasing the amount of organic nitrogen and diminishing, to the same extent, that of organic carbon. The necessity for using the front layer of oxide of copper is fully described in my "Water Analysis" (1st edition, p. 66, and 2nd edition, p. 65); and a sketch, showing the different layers in the combustion tube, is given on a page immediately following. When the combustion process is conducted in accordance with the directions there given, it yields results which, as tested by duplicate analyses, are in at least as close agreement as any obtainable in other direct organic determinations.—I am, etc.,

E. FRANKLAND.